

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Stoyanov et. al.

Attorney Docket No. 25384

Application No. 10/815,206

Group Art Unit: 1731

Filed: 03/31/2004

Examiner: Cordray, Dennis R.

Title: Individualized Intrafiber Crosslinked Cellulosic Fiber With Improved Brightness and Color

DECLARATION OF ANGEL STOYANOV PURSUANT TO § 37 C.F.R. § 1.132

Federal Way, WA,

October 4, 2006

TO THE COMMISSIONER OF PATENTS:

I, Angel Stoyanov, declare and state as follows:

1. I am currently employed by the Weyerhaeuser Company as a Scientist and since 1998 have worked exclusively on crosslinking of cellulosic fibers.

2. I received my Bachelor of Science and my Master Of Science from the University of Chemical Technology and Metallurgy at Sofia, Bulgaria, in 1980 and 1981, respectively. After graduation my work history is as follows:

I was a Research Assistant from 1982 to 1986 and an Assistant Professor from 1986 to 1994 at the University of Chemical Technology and Metallurgy at Sofia, Bulgaria. From 1990 to 1991 I worked under a Fulbright scholarship at the University of Washington, Seattle, WA, and completed all graduate courses for a Ph. D. in 1996. From

1996 to 1998 I conducted research for my Ph. D. and held various teaching positions in the Department of Engineering at the University of Washington.

3. I have read and am familiar with the Hansen et al patent US Patent No. 6,340,411

4. Hansen et al state in the '411 patent that initial application of the binder on high bulk fibers preferably occurs after the curing step, particularly if the binder is capable of functioning as a crosslinking material. Hansen then states that specific binders that can also crosslink are polyols, polyaldehydes, polycarboxylic acids and polyamines. If such binders are present during curing, the binder will be consumed during the curing step to form covalently crosslinked bonds. When this occurs, the binder is no longer available for hydrogen bonding or coordinate covalent bonding, and particle binding to fibers is ineffective, column 34, line 1-13.

5. Tests were undertaken to determine if polyols indeed act as crosslinking agents with cellulose. Accordingly, I planned and supervised experiments which were carried out by my technician Derik Rieger.

6. Exhibit A shows the experimental design for the tests. All samples were cured at 171°C for 7 minutes. The acronyms are as follows: COP, chemical on pulp (CF416 pulp from Weyerhaeuser Co.); SHP, sodium hypophosphite; CA, citric acid; SOR, sorbitol; and XYL, xylitol. Exhibit B shows the addition levels for the various reagents; Exhibit C gives the procedure, Exhibit D shows the results of brightness testing by TAPPI T 525 om-02 and Exhibit E, the FAQ wet bulk results determined by the procedure in the application. The Hunter color values were determined by TAPPI T 1231 sp 98. Whiteness Index, $WI_{(CDM-L)}$, was calculated from the formula, $WI_{(CDM-L)} = (L - 3b)$.

7. The results are summarized in Table 1.

Table 1

Fiber Properties

Sample	Wt. % on Dry Fiber				FAQ Wet Bulk, cc/g	ISO Brightness %	Hunter Color			W _I (CDM-L)
	CA	SHP	Sorbitol	Xylitol			L	a	b	
A	0	0	0	0	11.59	82.7	94.9	-0.83	5.58	78.16
B	0	2	0	0	12.26	82.8	95.0	-0.83	5.58	77.87
C	8	2	0	0	18.48	78.5	94.7	-2.02	8.67	68.69
D	8	2	2	0	18.29	83.7	95.3	-1.41	5.53	78.71
E	8	2	6	0	17.05	85.4	95.7	-1.23	4.80	81.3
F	8	2	0	2	18.18	84	95.6	-1.45	5.7	78.50
G	8	2	0	6	16.83	85.8	95.7	-1.21	4.53	82.10
H	0	2	2	0	11.43	82.3	94.8	-0.88	5.81	77.37
I	0	2	6	0	11.10	81.4	94.4	-0.81	5.96	76.52
J	0	2	0	2	11.27	80.5	94.1	-0.78	6.20	75.50
K	0	2	0	6	10.76	79.8	93.3	-0.76	5.60	76.50

8. It is well recognized by those skilled in the art of crosslinked fibers that an increase in FAQ wet bulk, relative to an untreated control, reflects that fibers have been crosslinked.

9. Sample A is a control and Sample B is the pulp with 2 percent by dry weight sodium hypophosphite; FAQ wet bulk values are 11.59 and 12.26 cc/g, respectively, and $WI_{(CDM-L)}$ values are 78.16 and 77.87, respectively. When pulp is treated with citric acid and sodium hypophosphite, Sample C, FAQ wet bulk is 18.48 cc/g and the Whiteness Index is 68.69. When pulp is treated with citric acid, sodium hypophosphite and sorbitol, a polyol, at the 2 and 6 percent by weight level of sorbitol on pulp, Samples D and E, respectively, FAQ wet bulk is significantly increased to 18.29 and 17.05 cc/g, respectively. The Whiteness Index of Samples D and E, also increased to 78.71 and 81.30, respectively. However, when pulp is treated only with sodium hypophosphite and two different levels of sorbitol, 2 and 6 percent by weight, Samples H and I, there is no increase in FAQ wet bulk; Whiteness Index, decreased relative to the control pulp and the pulp sample with only sodium hypophosphite, Samples A and B, respectively.

When pulp is treated with citric acid, sodium hypophosphite and xylitol, a polyol, at the 2 and 6 percent by weight level of xylitol on pulp, Samples F and G, respectively, FAQ wet bulk is significantly increased to 18.18 and 16.83 cc/g, respectively. The Whiteness Index of Samples F and G, also increased to 78.50 and 82.10, respectively. However, when pulp is treated only with sodium hypophosphite and two different levels of xylitol, 2 and 6 percent by weight, Samples J and K, there is no increase in FAQ wet bulk; Whiteness Index $WI_{(CDM-L)}$, decreased relative to the control pulp and the pulp with only sodium hypophosphite, Samples A and B, respectively.

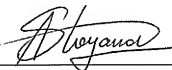
10. Based on the fact that there is no increase in FAQ wet bulk when pulp is treated only with sodium hypophosphite and sorbitol, or only with sodium hypophosphite and xylitol, it is my opinion that the polyol, sorbitol, and the polyol, xylitol, do not crosslink with cellulose.

11. In accordance with accepted Patent Office Practice, the dates in the laboratory notebook pages presented in Exhibits A- E have been redacted.

12. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

Respectfully submitted,

Date 10/4/06

A handwritten signature in black ink, appearing to read "A. Stoyanov", is written over a horizontal line.

Angel Stoyanov

EXHIBIT A

Project No. _____
 Book No. 14680

TITLE Exp # 145 Solutions

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From Page No.

Weyerhaeuser Confidential

Patent Action

Due Date: 8/4/2006

Title: Experiment # 145: CA + Polyols for Patent action

Objective(s): Investigate whether polyols will be involved in crosslinking of cellulose fibers under the conditions used for esterification of cellulose with CA

Materials:

- Pulp: CF416 - 99%
- Sample size: 20 g
- XLinker: CA
- Catalyst: SHP 99
- Polyols: Sorbitol (Sorbindex) and Xylitol (Xylidex) 98%
- Fiberizer: 6" pad former
- Dispatch oven
- Metal baskets for curing

Experimental Design:

Sample ID	Chemistry	XLinker	SHP	Polyol		Cure Temp.	Cure time
				Sorbitol	Xylitol		
		(% COP)	(% COP)	(COP)		(°F)	(min.)
A	Blank	0	0	0	0	340	7
B	Pulp+SHP	0	2	0	0	340	7
C	CA+SHP	8	2	0	0	340	7
D	CA+SHP+SOR	8	2	2	0	340	7
E	CA+SHP+SOR	8	2	6	0	340	7
F	CA+SHP+XYL	8	2	0	2	340	7
G	CA+SHP+XYL	8	2	0	6	340	7
H	SHP+SOR	0	2	2	0	340	7
I	SHP+SOR	0	2	6	0	340	7
J	SHP+XYL	0	2	0	2	340	7
K	SHP+XYL	0	2	0	6	340	7

Procedure:

1. Weigh the sample 20 g (odb);
2. Apply the crosslinking solution using the usual syringe method;
3. Leave the samples overnight in a sealed plastic bags;
4. Use the 6" pad former for fluffing (50% consistency);
5. Cure the samples in the Dispatch V Series oven;
6. Store the cured fibers in a plastic bag.

Testing:

1. AFAQ Wet Bulk at 0.6 kPa
2. Brightness/Color

Witnessed & Understood by me, _____ Date _____

Invented by _____

Recorded by _____

Date

8/1/06

EXHIBIT B

TITLE Exp# 145 solutionsProject No. _____
Book No. 14680

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From Page No. _____

Exp# 145:CA+ Polyols for patent actionDate: 8/1/2006Pulp CF416

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
A	CA	0	20	100	0.000	
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	0	20	1.20	0.000	

pH 7.15

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
B	CA	0	20	100	0.000	
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.483

pH 7.06

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
C	CA	8	20	100	1.600	1.597
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.482

pH 1.96

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
D	CA	8	20	100	1.600	1.603
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.477
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Sorbitol	2	20	100	0.400	0.401

pH 1.91

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
E	CA	8	20	100	1.600	1.603
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.470
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Sorbitol	8	20	100	1.200	1.202

pH 1.93

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
F	CA	8	20	100	1.600	1.605
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.480
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Xylitol	2	20	100	0.400	0.400

pH 1.92

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
G	CA	8	20	100	1.600	1.601
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.481
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Xylitol	8	20	100	1.200	1.191

pH 1.94

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
H	Sorbitol	2	20	100	0.400	0.399
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.485

pH 4.73

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Witnessed & Understood by me, _____

Date _____

Invented by D. J. P. J. P.

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Date _____

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EXHIBIT B

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Project No.

Book No. 19680

TITLE Exp # 145 Solution NATA

From Page No. ..

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
I	Sorbitol	6	20	100	1.200	1.202
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.482

pH 4.72

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
J	Xylitol	2	20	100	0.400	0.401
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.489

pH 4.75

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
K	Xylitol	6	20	100	1.200	1.199
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.484

pH 4.75

To Page No.

Witnessed & Understood by me,

Date

Invented by

Recorded by

Date

5/1/06

EXHIBIT C

 TITLE Exp#145: CA + Polyols for Patent actn Project No. _____
 Book No. 1960

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8/106

CF410 pulp used 20g O.D. @ 94% consistency = 21.28g pulp

(TARGET weight(g)) ← Actual weight(g) Pulp solution(g)

21.28g

21.24

40.88

21.15

41.06

21.21

41.15

21.38

41.26

21.34

41.31

21.23

41.33

21.37

41.17

21.15

41.03

21.34

41.13

21.25

40.98

21.22

41.13

- Prepared solutions on 8/106
 - applied to sheets
- Fiberized on 8/206 - Visual on fibers appears to be no different between samples, pre-curing.
- Samples air dry on table top for 4 hours before curing.
- Samples cured @ 340° for 7 min each on 8/206
- Samples placed in Soxhlet room before FAQ testing, 8/206
- TESTED: Brightness & color on 8/306
- FAQ TESTER in mezzanine NOT used after many controls would not comb into spec. ON 8/306
 - 8/106 FAQ TESTER in 116 was used

Witnessed & Understood by me, _____

Date _____

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Recorded by _____

Date _____

8/406

To Page No. _____

EXHIBIT D

Project No. _____
Book No. 14680TITLE EXPT 145 Brightness Results

From Page No. _____

Exp#	Sample#	side	position	Operator	TEST DATE	BRIGHTNESS	R(X)	R(Y)	R(Z)	X	Y	Z	L	a
145	A	a	1	D	08/03/06	82.44	91.01	89.69	82.21	87.52	89.60	97.2	94.7	-0.84
145	A	a	2		08/03/06	82.42	91	89.65	82.18	87.5	89.65	97.16	94.69	-0.8
145	A	a	3		08/03/06	82.39	90.98	89.64	82.14	87.48	89.64	97.12	94.68	-0.82
145	A	b	1		08/03/06	83.03	91.79	90.41	82.78	88.24	90.41	97.85	95.08	-0.81
145	A	b	2		08/03/06	83.05	91.8	90.44	82.78	88.25	90.44	97.88	95.1	-0.85
145	A	b	3		08/03/06	83.04	91.77	90.42	82.78	88.23	90.42	97.87	95.09	-0.85
Average						82.7	91.4	90.0	82.5	87.9	90.0	97.5	94.9	-0.8
StdDev						0.3	0.4	0.4	0.3	0.4	0.4	0.4	0.2	0.0
145	B	a	1	D	08/03/06	81.85	91.13	89.58	81.58	87.49	89.68	96.45	94.7	-0.88
145	B	a	2		08/03/06	81.87	91.05	89.58	81.42	87.39	89.58	96.27	94.65	-0.87
145	B	a	3		08/03/06	81.87	91.07	89.59	81.4	87.41	89.59	96.24	94.65	-0.87
145	B	b	1		08/03/06	83.8	92.16	90.92	83.57	88.99	90.92	98.81	95.35	-0.91
145	B	b	2		08/03/06	83.82	92.2	90.94	83.57	88.72	90.94	98.81	95.36	-0.88
145	B	b	3		08/03/06	83.79	92.15	90.89	83.55	88.68	90.89	98.79	95.34	-0.87
Average						82.8	91.8	90.3	82.5	88.1	90.3	97.6	95.0	-0.9
StdDev						1.1	0.6	0.7	1.2	0.7	0.7	1.4	0.4	0.0
145	C	a	1	D	08/03/06	78.52	91.12	89.54	77.97	86.77	89.54	92.19	94.83	-1.08
145	C	a	2		08/03/06	78.54	91.12	89.56	77.99	86.77	89.56	92.2	94.83	-1.08
145	C	a	3		08/03/06	78.59	91.19	89.63	78.02	86.83	89.63	92.25	94.87	-1.02
145	C	b	1		08/03/06	78.29	91.2	89.59	77.72	86.79	89.59	91.89	94.85	-2.03
145	C	b	2		08/03/06	78.61	91.57	89.93	78.02	87.13	89.93	92.24	94.83	-2.02
145	C	b	3		08/03/06	78.07	91.53	89.92	78.07	87.11	89.92	92.31	94.83	-2.04
Average						78.5	91.3	89.7	78.0	86.9	89.7	92.2	94.7	-2.0
StdDev						0.1	0.2	0.2	0.1	0.2	0.2	0.1	0.1	0.0
145	D	a	1	D	08/03/06	83.84	91.97	91.37	83.47	93.52	91.05	98.68	95.42	-1.46
145	D	a	2		08/03/06	84.11	92.19	91.28	83.7	93.74	91.28	98.98	95.54	-1.48
145	D	a	3		08/03/06	84.26	92.33	91.37	83.88	93.88	91.37	99.15	95.59	-1.39
145	D	b	1		08/03/06	83.28	91.33	90.38	82.88	87.9	90.38	98	95.07	-1.38
145	D	b	2		08/03/06	83.35	91.41	90.45	82.94	87.99	90.45	98.06	95.1	-1.37
145	D	b	3		08/03/06	83.5	91.52	90.59	83.09	88.09	90.59	98.24	95.18	-1.42
Average						83.7	91.8	90.9	83.3	88.4	90.9	98.5	95.3	-1.4
StdDev						0.4	0.4	0.4	0.4	0.4	0.4	0.5	0.2	0.0
145	E	a	1	D	08/03/06	85.07	92.18	91.39	84.78	89.94	91.39	100.23	95.6	-1.28
145	E	a	2		08/03/06	85.02	92.57	91.75	85.19	89.83	91.75	100.72	95.78	-1.22
145	E	a	3		08/03/06	85.63	92.63	91.8	85.28	89.39	91.8	100.81	95.81	-1.19
145	E	b	1		08/03/06	85.11	92.18	91.37	84.81	89.83	91.37	100.27	95.59	-1.28
145	E	b	2		08/03/06	85.34	92.42	91.8	85	89.17	91.8	100.5	95.71	-1.23
145	E	b	3		08/03/06	85.7	92.69	91.88	85.36	89.48	91.88	100.92	95.86	-1.22
Average						85.4	92.4	91.6	85.1	89.2	91.6	100.6	95.7	-1.2
StdDev						0.3	0.2	0.2	0.2	0.2	0.2	0.3	0.1	0.0
145	F	a	1	D	08/03/06	83.8	92.07	91.08	83.22	88.55	91.08	98.39	95.44	-1.45
145	F	a	2		08/03/06	83.91	92.35	91.34	83.49	88.62	91.34	98.71	95.57	-1.43
145	F	a	3		08/03/06	83.94	92.38	91.39	83.49	88.85	91.39	98.71	95.8	-1.48
145	F	b	1		08/03/06	83.99	92.24	91.3	83.0	88.76	91.3	98.65	95.55	-1.47
145	F	b	2		08/03/06	84.17	92.4	91.43	83.73	88.91	91.43	99	95.62	-1.43
145	F	b	3		08/03/06	84.08	92.31	91.36	83.69	88.83	91.36	98.95	95.59	-1.48
Average						84.8	92.3	91.3	83.5	88.8	91.3	98.8	95.6	-1.5
StdDev						0.2	0.1	0.1	0.2	0.1	0.1	0.2	0.1	0.0
145	G	a	1	D	08/03/06	85.64	92.26	91.54	85.38	90.12	91.54	100.82	95.68	-1.23
145	G	a	2		08/03/06	86.08	92.65	91.9	85.74	89.5	91.9	101.38	95.89	-1.17
145	G	a	3		08/03/06	86.04	92.67	91.89	85.74	89.51	91.89	101.37	95.86	-1.14
145	G	b	1		08/03/06	85.86	92.46	91.71	85.53	89.31	91.71	101.13	95.77	-1.19
145	G	b	2		08/03/06	85.86	92.3	91.55	85.29	89.14	91.55	100.85	95.68	-1.21
145	G	b	3		08/03/06	85.47	92.13	91.43	85.18	88.86	91.43	100.69	95.62	-1.3
Average						85.8	92.4	91.7	85.5	89.3	91.7	101.1	95.7	-1.2
StdDev						0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.1	0.1
145	H	a	1	D	08/03/06	82.22	91.17	89.78	81.93	87.59	89.78	96.87	94.75	-0.88
145	H	a	2		08/03/06	82.22	91.15	89.75	81.93	87.57	89.75	96.87	94.74	-0.85
145	H	a	3		08/03/06	82.17	91.09	89.72	81.87	87.51	89.72	96.79	94.72	-0.9
145	H	b	1		08/03/06	82.43	91.38	89.97	82.12	87.78	89.97	97.09	94.85	-0.88
145	H	b	2		08/03/06	82.35	91.31	89.93	82.05	87.72	89.93	97.01	94.83	-0.86
145	H	b	3		08/03/06	82.26	91.29	89.9	81.97	87.69	89.9	96.91	94.81	-0.89
Average						82.3	91.2	89.8	82.0	87.8	89.8	96.9	94.8	-0.9
StdDev						0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.0

Dr. Holte

Witnessed & Understood by me, _____

Date _____

Invented by _____

Recorded by _____

Date _____

8/10/06

To Page No. _____

EXHIBIT D

TITLE Exp# 145 Brightness ResultsBook No. 14600

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From Page No.

b	I'	a'	b'	HUNTER WSTMICIE V. CIE TINT									
5.52	95.87	-0.81	5.51	0	0	575.00	5.25	89.89	59.8	64.42	-1.93		
5.52	95.85	-0.77	5.51	0	0	575.15	5.26	89.85	59.77	64.38	-1.99		
5.54	95.85	-0.79	5.54	0	0	575.11	5.28	89.84	59.94	64.27	-1.97		
5.63	96.17	-0.78	5.62	0	0	575.18	5.34	90.41	59.81	64.74	-2.03		
5.63	96.18	-0.82	5.62	0	0	575.07	5.34	90.44	59.83	64.77	-1.97		
5.62	96.17	-0.82	5.61	0	0	575.05	5.33	90.42	59.80	64.79	-1.96		
5.6	96.0	-0.8	5.6	0.0	0.0	575.1	5.3	90.0	59.8	64.6	-2.0		
0.1	0.2	0.0	0.1	0.0	0.0	0.0	0.0	0.4	0.1	0.2	0.0		
5.99	95.87	-0.85	5.99	0	0	575.12	5.71	89.68	57.27	62.21	-2.14		
6.03	95.82	-0.84	6.04	0	0	575.17	5.76	89.58	56.96	61.89	-2.19		
6.06	95.83	-0.84	6.06	0	0	575.18	5.79	89.59	56.83	61.77	-2.21		
5.4	96.38	-0.87	5.37	0	0	574.8	5.08	90.02	61.51	66.42	-1.72		
5.41	96.39	-0.85	5.38	0	0	574.87	5.09	90.04	61.48	66.39	-1.73		
5.39	96.37	-0.84	5.35	0	0	574.89	5.07	90.09	61.53	66.43	-1.77		
5.7	96.1	-0.8	5.7	0.0	0.0	575.0	5.4	90.3	59.3	64.2	-2.0		
0.3	0.3	0.0	0.4	0.0	0.0	0.2	0.4	0.7	2.5	2.4	0.2		
8.96	95.81	-1.92	8.69	0	0	573.8	8.17	89.54	43.27	49.58	-1.85		
8.96	95.81	-1.94	8.69	0	0	573.75	8.17	89.50	43.27	49.59	-1.8		
8.98	95.84	-1.96	8.71	0	0	573.72	8.19	89.63	43.2	49.57	-1.78		
8.79	95.82	-1.97	8.92	0	0	573.79	8.39	89.59	42.12	48.57	-1.85		
8.79	95.97	-1.96	8.93	0	0	573.82	8.39	89.93	42.28	48.91	-1.91		
8.75	95.96	-1.97	8.88	0	0	573.77	8.34	89.92	42.53	49.12	-1.85		
8.7	95.9	-2.0	8.8	0.0	0.0	573.8	8.3	89.7	42.8	49.2	-1.8		
0.1	0.1	0.0	0.1	0.0	0.0	0.6	0.6	0.1	0.2	0.5	0.4		
5.58	96.43	-1.41	5.54	0	0	573.29	5.14	91.05	61.73	65.77	-0.88		
5.55	96.52	-1.41	5.52	0	0	573.28	5.12	91.28	60.97	66.09	-0.88		
5.5	96.50	-1.33	5.47	0	0	573.49	5.06	91.37	61.32	66.44	-0.97		
5.52	96.15	-1.33	5.5	0	0	573.5	5.12	90.38	60.41	65.23	-0.98		
5.52	96.16	-1.32	5.51	0	0	573.54	5.13	90.45	60.43	65.27	-1.01		
5.51	96.24	-1.38	5.49	0	0	573.37	5.11	90.59	60.8	65.49	-0.9		
5.5	96.3	-1.4	5.5	0.0	0.0	573.4	5.1	90.9	60.7	65.7	-0.9		
0.0	0.2	0.0	0.0	0.0	0.0	0.1	0.8	0.4	0.4	0.5	0.1		
4.84	96.57	-1.24	4.8	0	0	573.26	4.44	91.39	64.95	69.55	-0.73		
4.79	96.72	-1.17	4.76	0	0	573.43	4.4	91.75	65.51	70.16	-0.81		
4.78	96.74	-1.15	4.73	0	0	573.51	4.38	91.8	65.85	70.3	-0.84		
4.8	96.56	-1.22	4.78	0	0	573.29	4.4	91.37	65.14	69.7	-0.74		
4.82	96.85	-1.19	4.78	0	0	573.41	4.42	91.6	65.22	69.86	-0.8		
4.78	96.77	-1.18	4.71	0	0	573.39	4.36	91.88	65.79	70.45	-0.78		
4.8	96.7	-1.2	4.8	0.0	0.0	573.4	4.4	91.8	65.4	70.9	-0.8		
0.0	0.1	0.0	0.0	0.0	0.0	0.1	0.0	0.2	0.3	0.4	0.0		
5.77	96.44	-1.4	5.75	0	0	573.48	5.34	91.08	59.84	64.88	-1		
5.75	96.55	-1.38	5.73	0	0	573.52	5.33	91.34	59.91	65.21	-1.03		
5.78	96.57	-1.41	5.76	0	0	573.48	5.35	91.39	59.79	65.13	-1		
5.64	96.53	-1.42	5.61	0	0	573.33	5.2	91.3	60.51	65.71	-0.9		
5.64	96.59	-1.38	5.61	0	0	573.44	5.21	91.43	60.83	65.87	-0.96		
5.63	96.57	-1.43	5.61	0	0	573.3	5.19	91.38	60.82	65.83	-0.88		
5.7	96.5	-1.4	5.7	0.0	0.0	573.4	5.3	91.3	60.2	65.4	-1.0		
0.1	0.1	0.0	0.1	0.0	0.0	0.1	0.1	0.1	0.5	0.4	0.1		
4.52	96.83	-1.18	4.47	0	0	573.17	4.13	91.54	69.81	71.18	-0.64		
4.49	96.78	-1.12	4.44	0	0	573.35	4.11	91.9	67.38	71.72	-0.72		
4.49	96.78	-1.1	4.44	0	0	573.45	4.11	91.68	67.28	71.71	-0.76		
4.52	96.7	-1.14	4.47	0	0	573.31	4.13	91.71	67	71.41	-0.7		
4.57	96.63	-1.17	4.52	0	0	573.26	4.18	91.55	68.54	70.96	-0.69		
4.50	96.59	-1.25	4.54	0	0	572.97	4.19	91.43	68.35	70.75	-0.55		
4.5	96.7	-1.2	4.5	0.0	0.0	573.3	4.13	91.7	66.9	71.3	-0.7		
0.0	0.1	0.1	0.0	0.0	0.0	0.2	0.0	0.2	0.4	0.4	0.1		
5.8	95.91	-0.84	5.8	0	0	575.06	5.52	89.78	58.37	63.21	-2.03		
5.78	95.98	-0.82	5.77	0	0	575.12	5.5	89.78	58.49	63.3	-2.06		
5.8	95.98	-0.87	5.8	0	0	574.99	5.52	89.72	58.32	63.14	-1.99		
5.8	95.98	-0.84	5.79	0	0	575.07	5.51	89.97	58.55	63.45	-2.03		
5.82	95.97	-0.86	5.81	0	0	575.02	5.53	89.93	58.41	63.31	-2.01		
5.95	95.95	-0.86	5.85	0	0	575.04	5.57	89.9	58.19	63.1	-2.04		
5.8	95.9	-0.8	5.8	0.0	0.0	575.1	5.5	89.8	58.4	63.3	-2.0		
0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.1	0.0		

To Page No. _____

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8/10/06

EXHIBIT D

90

Project No.
Book No. 14660

TITLE Exp# 145 Brightness Results

From Page No.

Exp#	Sample#	side	position	Operator	TEST DATE	BRIGHTNESS	R(X)	R(Y)	R(Z)	X	Y	Z	L	S
145	I	a	1	D	08/03/06	81.45	90.64	89.18	81.19	87.03	89.16	96	94.43	-0.8
145	I	a	2		08/03/06	81.47	90.63	89.18	81.21	87.03	89.16	96.01	94.42	-0.79
145	I	a	3		08/03/06	81.38	90.56	89.09	81.11	86.05	89.09	95.9	94.39	-0.81
145	I	b	1		08/03/06	81.46	90.77	89.27	81.18	87.13	89.27	95.88	94.48	-0.81
145	I	b	2		08/03/06	81.38	90.71	89.21	81.07	87.08	89.21	95.85	94.45	-0.83
145	I	b	3		08/03/06	81.38	90.73	89.21	81.08	87.08	89.21	95.86	94.45	-0.79
					Average	81.4	90.7	89.2	81.1	87.0	89.2	95.9	94.4	-0.8
					StdDev	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.0	0.0
145	J	a	1	D	08/03/06	80.46	90.08	88.51	80.19	88.39	88.51	94.81	94.08	-0.78
145	J	a	2		08/03/06	80.47	90.05	88.47	80.2	88.37	88.47	94.82	94.06	-0.77
145	J	a	3		08/03/06	80.33	89.95	88.38	80.07	88.27	88.38	94.67	94.01	-0.78
145	J	b	1		08/03/06	80.72	90.39	88.78	80.45	88.68	88.78	95.12	94.22	-0.78
145	J	b	2		08/03/06	80.59	90.27	88.59	80.3	88.57	88.68	94.84	94.17	-0.77
145	J	b	3		08/03/06	80.48	90.19	88.8	80.2	88.48	88.5	94.82	94.13	-0.79
					Average	80.5	90.2	88.6	80.2	88.5	88.6	94.9	94.1	-0.8
					StdDev	0.1	0.2	0.1	0.1	0.1	0.1	0.2	0.1	0.0
145	K	a	1	D	08/03/06	80.24	88.94	87.58	80	85.46	87.58	94.59	93.58	-0.8
145	K	a	2		08/03/06	80.3	88.99	87.59	80.08	85.51	87.59	94.65	93.59	-0.75
145	K	a	3		08/03/06	80.29	88.87	87.57	80.05	85.5	87.57	94.64	93.58	-0.74
145	K	b	1		08/03/06	79.49	87.99	86.63	79.25	84.57	86.63	93.7	93.07	-0.76
145	K	b	2		08/03/06	79.35	87.87	86.5	79.1	84.45	86.5	93.52	93	-0.75
145	K	b	3		08/03/06	79.33	87.88	86.5	79.08	84.43	86.5	93.5	93.01	-0.78
					Average	79.8	88.4	87.1	79.6	85.0	87.1	94.1	93.5	-0.8
					StdDev	0.5	0.6	0.6	0.5	0.6	0.6	0.6	0.3	0.0

D. Blalock

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To Page No.

Date

EXHIBIT D

TITLE Exp # 145 Brightness ResultsBook No. 14680

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From Page No. _____

Dr. Shuler

b	L*	a*	b*	HUNTER WSTM CIE V. CIE TINT							
5.91	95.65	-0.77	5.91	0.0	0	575.32	5.68	89.16	57.29	82	-2.25
5.89	95.65	-0.76	5.9	0	0	575.34	5.65	89.16	57.36	82.05	-2.26
5.92	95.62	-0.76	5.93	0	0	575.27	5.68	89.09	57.16	81.85	-2.22
6	95.69	-0.76	6.01	0	0	575.3	5.75	89.27	58.88	81.69	-2.27
6.04	95.67	-0.8	6.06	0	0	575.27	5.79	89.21	58.63	81.44	-2.27
6.02	95.67	-0.76	6.03	0	0	575.38	5.78	89.21	58.7	81.5	-2.33
6.0	95.7	-0.8	6.0	0.0	0.0	575.3	5.7	89.2	57.0	81.8	-2.3
6.1	95.7	-0.8	6.1	0.0	0.0	575.3	5.7	89.2	57.0	81.8	-2.3
6.19	95.37	-0.76	6.21	0	0	575.44	5.97	88.51	55.25	59.9	-2.45
6.18	95.38	-0.74	6.18	0	0	575.48	5.95	88.47	55.38	60	-2.47
6.19	95.32	-0.75	6.21	0	0	576.40	5.99	88.38	55.14	59.74	-2.47
6.19	95.49	-0.75	6.21	0	0	575.46	5.97	88.76	55.48	60.21	-2.46
6.23	95.45	-0.75	6.25	0	0	575.48	6.01	88.68	55.16	59.9	-2.49
6.25	95.41	-0.76	6.27	0	0	575.44	6.03	88.6	55	59.72	-2.48
6.2	95.4	-0.8	6.2	0.0	0.0	575.5	6.0	88.6	55.2	59.9	-2.5
6.0	95.4	-0.8	6.0	0.0	0.0	575.5	6.0	88.6	55.2	59.9	-2.5
5.00	94.98	-0.77	5.67	0	0	575.22	5.46	87.56	57.32	61.34	-2.1
5.63	94.99	-0.73	5.65	0	0	575.34	5.44	87.59	57.46	61.48	-2.17
5.63	94.98	-0.71	5.65	0	0	575.37	5.44	87.57	57.47	61.49	-2.19
5.54	94.98	-0.73	5.57	0	0	575.29	5.38	86.63	57.14	60.8	-2.12
5.57	94.53	-0.72	5.6	0	0	575.33	5.41	86.5	56.9	60.53	-2.15
5.59	94.53	-0.76	5.61	0	0	575.23	5.43	86.5	56.81	60.46	-2.1
5.6	94.8	-0.7	5.6	0.0	0.0	575.3	5.4	87.1	57.2	61.0	-2.1
6.0	94.8	-0.7	6.0	0.0	0.0	575.3	5.4	87.1	57.2	61.0	-2.1

To Page No. _____

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Date _____

8/10/09

EXHIBIT E

 Project No. _____
 Book No. 14660

 TITLE Exp# 145 FAQ Results

From Page No. _____

Test Date	Ref # or Sample Number	Hub Number	Hub Grade	Fluorizer Type	Full Cond	Mandate Date	Mandate Time	Operator Initial	Lab Name	Run Number	Dry Bulk cc/g	Dry Bulk 2.0kPa cc/g	Wick Time sec	Wick Rate mm/s	Wick Bulk cc/g	Wick Bulk 2.0kPa cc/g	Capacity g/g
04/02/00	exp#145	Albark	ch16	O	A	8/12/00		dr	Lab 116	1	40.84	24.30	2.6	8.55	3.95	11.74	12.92
04/02/00	exp#145	Albark	ch16	O	A	8/12/00		dr	Lab 116	2	38.57	23.17	2.4	10.65	8.63	11.49	11.59
04/02/00	exp#145	Albark	ch16	O	A	8/12/00		dr	Lab 116	3	40.82	24.32	2.5	10.82	8.70	11.05	12.08
04/02/00	exp#145	Albark	ch16	O	A	8/12/00		dr	Lab 116	4	40.11	23.96	2.97	10.77	8.18	11.02	11.91
04/02/00	exp#145	BIPup-H4	ch16	O	A	8/12/00		dr	Lab 116	1	40.04	24.26	2.4	11.29	10.40	12.19	12.82
04/02/00	exp#145	BIPup-H4	ch16	O	A	8/12/00		dr	Lab 116	2	40.94	24.92	2.1	12.90	10.52	12.25	12.82
04/02/00	exp#145	BIPup-H4	ch16	O	A	8/12/00		dr	Lab 116	3	40.88	24.20	2.6	10.4	10.48	12.32	12.55
04/02/00	exp#145	BIPup-H4	ch16	O	A	8/12/00		dr	Lab 116	4	40.92	24.20	2.37	11.53	10.49	12.26	12.44
04/02/00	exp#145	CBCA-H2	ch16	O	A	8/12/00		dr	Lab 116	1	41.91	25.20	2.9	10.80	15.21	10.18	10.40
04/02/00	exp#145	CBCA-H2	ch16	O	A	8/12/00		dr	Lab 116	2	44.62	26.38	3	10.98	10.79	18.74	18.65
04/02/00	exp#145	CBCA-H2	ch16	O	A	8/12/00		dr	Lab 116	3	42.03	25.61	3.2	10.03	15.50	10.55	10.82
04/02/00	exp#145	CBCA-H2	ch16	O	A	8/12/00		dr	Lab 116	4	42.85	25.76	3.03	10.92	15.53	10.49	10.59
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	1	43.7	28.16	3.1	10.49	15.53	18.48	18.68
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	2	44.97	28.78	3.3	10	15.59	18.48	18.53
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	3	41.78	25.29	2.9	10.70	14.89	17.8	18.08
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	4	43.38	28.00	3.1	10.42	15.54	18.28	18.23
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	1	41.80	25.22	3.4	10.87	15.0	16.75	16.8
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	2	42.38	25.01	2.8	11.13	14.38	17.25	17.61
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	3	42.9	25.30	2.9	10.94	14.36	17.13	17.37
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	4	42.27	25.29	2.7	11.48	14.14	17.05	17.26
04/02/00	exp#145	FCa-HBF	ch16	O	A	8/12/00		dr	Lab 116	1	44.41	28.67	3.2	10.27	15.59	16.42	16.57
04/02/00	exp#145	FCa-HBF	ch16	O	A	8/12/00		dr	Lab 116	2	45.1	28.94	3.9	10.9	16.52	16.03	16.19
04/02/00	exp#145	FCa-HBF	ch16	O	A	8/12/00		dr	Lab 116	3	42.68	25.8	2.9	10.58	15.38	18.1	18.42
04/02/00	exp#145	FCa-HBF	ch16	O	A	8/12/00		dr	Lab 116	4	43.98	25.97	3	10.72	15.23	16.16	16.30
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	1	41.71	24.94	2.8	11.8	13.73	15.49	15.66
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	2	44.18	25.67	2.8	11.95	14.31	17.67	17.17
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	3	43.06	25.48	2.7	11.43	14.12	15.94	16.05
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	4	42.97	25.28	2.63	11.84	14.09	18.33	18.23
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	1	38.72	23.04	2.2	11.05	8.96	11.17	11.89
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	2	41.33	23.08	2.3	11.41	10.91	11.82	11.82
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	3	42.1	24.28	2.1	12.09	9.96	11.49	12.00
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	4	41.03	23.86	2.2	11.86	9.84	11.43	11.84
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	1	42.8	24.51	2.4	11.13	9.75	11.17	11.39
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	2	42.1	23.89	2.5	10.24	9.37	10.91	11.36
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	3	43.91	24.61	2.7	9.87	9.99	11.23	11.55
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	4	42.9	24.17	2.53	10.41	9.6	11.1	11.43
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	1	41.78	24.52	2.1	12.17	9.77	10.91	11.49
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	2	42.42	23.84	2.4	11.82	10.21	11.58	11.96
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	3	43.7	24.88	2.4	11.19	9.88	11.38	11.71
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	4	42.63	23.98	2.3	11.48	9.15	11.27	11.72
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	1	42.63	23.1	2.2	11.05	9.5	11.04	11.33
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	2	42.05	22.27	2.1	11.97	8.82	10.4	10.88
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	3	42.74	23.42	2.3	11.13	9.43	10.96	11.1
04/02/00	exp#145	OCa-HSH	ch16	O	A	8/12/00		dr	Lab 116	4	41.81	22.95	2.2	11.42	9.29	10.76	11.18

M. Duke

To Page No. _____

Witnessed & Understood by me, _____

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Date _____

8/10/06